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Short - Abstract

A multidisciplinary Carnegie Mellon University (CMU) research team studied new high temperature magnetic materials. CMU studied existing bulk soft materials and on novel nanocrystalline magnets in parallel. A search for new hard and soft magnetic materials was Prototypes for bulk soft magnets were Fe-Co alloys. The effects of grain size, structural order, orientation, and texture were studied to maximize induction and permeability, and minimize hysteretic and eddy current losses at elevated temperatures. Investigation of nanocrystalline materials for soft magnetic applications proceeded on two fronts. The first was plasma synthesis of magnetic nanoparticles followed by the use of compaction techniques to form dense magnets. The second route involved nanocrystallization of amorphous precursors to produce exchange coupled magnetic nanocrystals. The first has led to synthesis of metallic nanoparticles, of new core shell structures and nanocrystalline ferrites. The second has led to the discovery of a new nanocrystalline soft magnetic material called HITPERM for which work is continuing for the use of these materials in power electronic applications. Co-containing 2:17, 1:12, and 3:29 - based permanent magnet materials were investigated as hard magnetic materials (to 400 °C). We investigated the fundamental properties of these alloy systems as well as processing protocols for engineering microstructures. Co-containing 3:29 phase magnets have extended the temperature range of these materials. Rapid solidification processing was used as a means to try to tailor microstructure.

Advanced Magnetic Materials for Aircraft Power Applications MURI FINAL REPORT Air Force Office of Scientific Research March 4, 2003

Carnegie Mellon University Final Report



Table of Contents

Table of Contents	2	
Abstract		
A. Brief History	4	
B. Senior Personnel	5	
C. Summary of Accomplishments for Research Performed during		
MURI No-cost Extension through August, 2002.	5	
D. Conclusions	5	
Appendix I: Final MURI Review Schedule – AFOSR 2001		
Appendix II: Publications Acknowledging MURI Support	9	
Acknowledgements	12	

For detailed information about results of the CMU MURI Program visit our Web site at http://neon.mems.cmu.edu/muri/

Advanced Magnetic Materials for Aircraft Power Applications

Carnegie Mellon University MURI - Final Report

Abstract

A multidisciplinary team of Carnegie Mellon University (CMU) researchers with substantial support from industrial, national and DOD laboratory collaborators were assembled to develop new materials with improved magnetic and mechanical properties at high temperature. Our CMU group worked on the refinement of existing soft bulk materials while conducting research on novel nanocrystalline magnets in parallel. The team also studied existing and new high temperature permanent magnetic materials for use in aircraft engine applications. Extensive microstructural and magnetic characterization facilities at CMU were used to characterize materials. A vigorous search for new magnetic materials, both hard and soft was conducted. We employed a group of undergraduate and graduate students and post-doctoral associates in pursuing our research goals. In addition, we worked closely with experts from industry and national laboratories and coordinated with Wright Patterson Air Force Base (WPAFB) to improve existing and develop new high temperature magnetic materials for aircraft applications.

Prototypes for bulk soft magnetic materials were Fe-Co alloys where we studied the effects of grain size, structural order, orientation, and texture so as to maximize induction and permeability, and minimize hysteretic and eddy current losses in the 550 - 600 °C temperature range. We examined LaCo₁₃ and other novel magnetic nitrides (Fe-nitride) as potential high induction, high T_c materials. We had a vigorous effort in investigating nanocrystalline magnetic materials for soft magnetic applications. This proceeded on two fronts. The first was plasma synthesis of magnetic nanoparticles followed by the use of compaction techniques to form dense magnets. The second route involved nanocrystallization of amorphous precursors to produce exchange coupled magnetic nanocrystals. The first has led to synthesis of metallic nanoparticles, of new core shell structures and nanocrystalline ferrites. The second has led to the discovery of a new nanocrystalline soft magnetic material called HITPERM for which work is continuing for the use of these materials in power electronic applications.

For hard magnetic materials capable of operating up to 400 °C we investigated Cocontaining 2:17; 1:12, and 3:29 - based permanent magnet materials. We investigated the fundamental properties of these alloy systems as well as processing protocals for engineering optimal microstructures for hard magnetic properties. Co-containing 3:29 phase magnets have extended the temperature range of these materials. Rapid solidification processing was used as a means to try to tailor microstructure and to harness anisotropy and translate it into coercivity.

A. Brief History

A multidisciplinary team of Carnegie Mellon University (CMU) researchers, listed below, with substantial support from industrial, national and DOD laboratory collaborators were assembled to develop new materials with improved magnetic and mechanical properties at high temperature. The Multidisciplinary University Research Award (MURI) was entitled Advanced Magnetic Materials for Aircraft Power Applications (F49620-96-1-0454), Michael E. McHenry, PI and was pursued through a no-cost extension until August, 2002. The CMU group worked on the refinement of existing soft bulk materials while conducting research on novel nanocrystalline magnets in parallel. They also studied existing and new high temperature permanent magnetic materials for use in aircraft engine applications.

The details of the research have been summarized in a series of MURI booklets provided at annual review meetings held jointly with the University of Delaware and other research groups working on ancillary problems. Appendix I list the agenda for the Final MURI Review held May 10, 2001. The research out put of the CMU MURI Program has been detailed in these review documents. The MURI program continued through September, 2002 under a no-cost extension during which time industrial technology transfer interactions were pursued with ABB Corporation and the Magnetics Division of Spang and Company. During this time additional MURI research was published and applications of materials developed in the MURI project explored. Appendix II contains an list of archival publications produced with MURI support or partial MURI support.

The CMU MURI invented a new class of nanocrystalline $(Fe_{1-x} Co_x)_{88}M_7B_4Cu$ (M = Nb, Zr, Hf) soft magnetic materials called HITPERM. The excellent soft magnetic properties and magnetization that persists to elevated temperatures (up to ~ 600 C) as well as large magnetic inductions make these materials potential candidates for aircraft power electronic applications. The goals of the MEA include operation of aircraft engines at elevated temperatures. The CMU-ABB collaboration provided capital for facilitating development of these exciting alloys at CMU.

CMU researchers also developed new 3:29 phase permanent magnet materials. The 3:29 phase was shown to have large Curie temperatures suggesting its promise as a high temperature permanent magnet material. In collaboration with IIT Madras, CMU developed 3:29 magnets $(Pr_3(Fe_{1-x}Co_x)27.5Ti_{1.5} (x = 0, 0.1, 0.2, 0.3 \text{ and } 0.4)$ with up to 40% Co substitution for Fe. These exhibited large increases in Curie temperatures as compared with the Fe only phase.

CMU researchers developed of methods to prepare highly monodisperse FeCo nanoparticles for compaction, using a solution chemistry approach in nonaqueous solvents. They were able to tune the particle size with this synthesis between 15 and 900 nm, though for the smallest particle sizes, the surfaces of the equiaxed particles were somewhat rougher. They found that while the particle size could be varied dramatically, the average grain size ranged between 15 and 40 nm. The large particles prepared were only composites of smaller ones.

The MURI efforts had many notable educational and training achievements. These included the development of multidisciplinary course called "Structure and Applications of Magnetic Materials". Graduate students fully or partially supported by the MURI program have continued in important roles in government, academia and industry. Of particular note among these students are:

Zafer Turgut Researcher at Wright Patterson Air Force Base through

UES Inc., 4401 Dayton-Xenia Rd. Dayton, OH 45432.

Matthew Willard NRC Postdoctoral Associate; Naval Research Laboratory

John Henry Scott

Member of Technical Staff, NIST

Roberta Sutton

Faculty, Division of Materials. The University of Queensland.

Amy Hsiao

Faculty Union College, Schnectady, NY.

Krishna Chowdary

Instructor, Bucknell University.

Many undergraduate research projects were supported by the MURI program including senior projects. Of particular note in terms of the subsequent accomplishments of these students are:

Katy Gallagher

Ph.d Trinity College, Dublin; Applied Physics, now NRC

Postdoctoral Associate; Naval Research Laboratory.

Colin A. Ashe

NSF graduate student fellowship Ph.d Program. MIT MSE.

Daniel Schmidt

Ph.d Program. Cornell MSE.

Frank Johnson Mitra Taheri Ph.d Program. CMU MSE. Ph.d Program. CMU MSE.

Asumini Kasule

M.S. Stanford, MSE.

Joshua McKinley

Ph.d Program. University of Michigan, MSE.

Nathan Browand

Cambridge, Applied Mathematics.

Cindy Dennis

Oxford, Physics

Elise Selinger

Technical Specialist - Foundry Team; Stryker Howmedica Osteonics

Rebecca Berngartt

M. S. Program, Lincoln University; Canterbury, New Zealand

B. Senior Personnel

B.1. Investigators: Michael E. McHenry (PI), David E. Laughlin, Sara A. Majetich, Henry R. Piehler, Anthony Rollett, W. Edward Wallace, Meiqing Huang, Shaovan Chu, Anit Giri

B.2. Consultant: Fred Rothwarf

C. Summary of Accomplishments for Research Performed during MURI No-cost Extension

Industrial technology transfer interactions were pursued with ABB Corporation and the Magnetics Division of Spang and Company.

CMU participated in a collaborative research program with ABB in the area of advanced magnetic materials for high temperature applications. This was a technology transfer effort to the Multidisciplinary University Research Award (MURI) entitled Advanced Magnetic Materials for Aircraft Power Applications (F49620-96-1-0454), Michael E. McHenry, PI and its no-cost extension until August, 2002. The focus of the work was on the development of new high temperature soft and hard magnetic materials. ABB-CMU collaborative research focused on scaling-up rapid solidification processing efforts to produce precursors to HITPERM soft magnets and 3:29 phase hard magnetic materials.

CMU researchers continued rapid solidification processing to produce amorphous precursors to 3:29 phase permanent magnet materials. Rapid solidification and high energy ball milling were used in attempts to tailor the microstructures of materials to improve coercivities, a key materials property of the hard magnets. Arc melt spinning equipment was used, in collaboration with the Univ. of Pittsburgh to produce 3:29 phase materials. While promising intrinsic magnetic properties were demonstrated for 3:29 phase magnets, efforts to develop a nanostructure yielding large coercivities were unsuccessful. For both rapid solidification

processing and ball milling a decomposition of the 3:29 phase was observed to yield significant amounts of an α -FeCo soft magnetic phase that it was concluded limits their coercivity.

CMU scaled-up production of HITPERM magnet production using rapid solidification processing equipment both for soft and hard magnetic materials. The CMU/ABB collaboration provided funds for post-doctoral support and graduate student support in process development using this equipment as well as properties assessment and microstructural characterization. Pressurized quartz crucible jet casting equipment was used for production of 0.25 inch amorphous precursors to HITPERM materials. Process development for wider ribbons, namely planar flow casting was attempted. Planar flow casting crucibles were designed and iterations of PFC trials were performed. Ribbons were wound into cores and AC losses were studied. During the CMU/ABB collaboration, HITPERM was been shown to have superior soft magnetic properties including permeabilities in excess of 1000, and losses less than that of Supermendur. In the collaboration we identified a property window ($\mu > 1000$, B > 1.7 T, f > 200 kHz, T up to 300 C), appropriate for DC/DC converter applications of interest to the Air Force.

The development of processing facilities for scale-up of amorphous and HITPERM alloys was also used in the industrial interactions with the Magnetics Division of Spang and Company (Magnetics). Magnetics, has interest in developing amorphous Fe-based alloys for use in tape cores, powder cores and stacked laminates. New work was initiated involving optimization of Fe-based metallic glass compositions on CMUs melt spinner, prior to installation of a production unit at Magnetics. These materials also hold considerable promise for magnetic power electronic devices.

CMU and Magnetics have entered into a TIA-Consortium and are currently partners in a Dual Use Science and Technology Program funded by Wright Patterson Air Force base. Because of reduced size and weight it is anticipated that amorphous and nanocrystalline alloys can be formed into magnetic components, including powder and tape wound cores that will operate to 100 KHz and above, with the potential to replace and outperform conventional ferrites in certain applications for aircraft DC/DC converter circuits. We anticipate operational temperatures > 300 C will be possible. With further research to increase resistivity in HITPERM precursor materials we expect that we may be able to extend their operational frequency to > 200 KHz. The ultimate goal of the DUST program is to develop magnetic materials to achieve high temperature operation to > 300 C. Our partner, Magnetics, has interest in developing amorphous Fe-based alloys for use in tape cores, powder cores and stacked laminates. Joint work includes optimizing Fe-based metallic glass compositions on our melt spinner, prior to installation of a production unit at Magnetics, understanding how to make nanocomposites by compaction, how to relate their properties to those of the constituent nanoparticles, how to optimize their magnetic properties.

Another highlight of work performed in the NCE to the MURI was the development of methods to prepare highly monodisperse FeCo nanoparticles for compaction, using a solution chemistry approach in nonaqueous solvents. We were able to tune the particle size with this synthesis between 15 and 900 nm, though for the smallest particle sizes, the surfaces of the equiaxed particles were somewhat rougher. We found that while the particle size could be varied dramatically, the average grain size ranged between 15 and 40 nm. The large particles prepared were only composites of smaller ones. Several compaction methods were investigated for similar samples: cold and hot isostatic pressing, magnetic compaction, and plasma pressure compaction, where a conducting arc is struck through the sample and it is rapidly compressed. Cold isostatic compaction yielded only 60% of full density. Hot isostatic compaction was able to

generate fully dense samples, but this often occurred at the expense of significant grain growth, which was detrimental to the desired magnetic properties. The magnetic compaction technique is in principle ideal, since the compaction time is the shortest (45 microseconds). However, in its current form there are insufficient precautions taken to prevent sample oxidation, and the compaction pressure used was too low, yielding only 50% of full density. Our best results were obtained with the plasma pressure compaction method, with routine densities over 90% and often approaching 100%, particularly with milled materials. The company we collaborated with on this method was Materials Modification, Inc., of Fairfax, VA.

The AC magnetic properties of the composites were investigated in toroidal samples as a function of temperature. The highest permeabilities of the compacted FeCo samples were still fairly low, 100-200, but roll-off frequencies as high as 700,000 Hz were measured. Models of the temperature-dependent properties suggest that the magnetic coupling between grains is at least an order of magnitude lower than that within the grains, and this is the main target for improvement.

D. Conclusions and Recommendations

FeCo nanoparticles were compacted by a variety of methods reported on in the CMU thesis of Zafer Turgut. Since Zafer's thesis the MURI group pursued compaction of FeCo and ferrite particles in collaboration with IAP Research (Bhanu Celluri) using a magnetic compaction technique and with Teodor Veres of the Canadian NRC as well as the efforts described above. Again these techniques have yet to provide full densities. In soft magnetic materials, full density is imperative, and even in samples compacted to 99% of full density the residual porosity is enough to degrade the magnetic properties. While these techniques may be useful for nanocomposite permanent magnets, and low permeability ferrite materials, they have yet to provide the densities required for high permeability soft magnetic materials applications.

At this point in time the most attractive soft magnetic properties have been documented for the nanocomposite magnets produced by nanocrystallization of amorphous precursors. While these materials are brittle when fully nanocrystallized, they display improved ductility on partial nanocrystallization. There have also been reports of improved ductility in hot extruded nanocomposite materials. While a variety of soft magnetic nanocomposites have been produced, some with excellent soft magnetic properties, it is not felt that these materials are competitive with bulk FeCo laminates in terms of combined magnetic and mechanical properties. These materials do have promise in high temperature electronic inductive components that are of interest to the Air Force and are continuing to be pursued for these applications.

3:29 phase magnets studied by CMU researchers were determined to have promising intrinsic magnetic properties. Nanostructured 3:29 phase materials were produced by bll milling and rapid solidification. These nanostructures did not yielding large coercivities due to a decomposition reaction that yielded significant amounts of an α -FeCo soft magnetic phase that it was concluded limits their coercivity.

Appendix I: Final MURI Review Schedule - AFOSR 2001

MURI on Advanced Magnetic Materials Final Program Review May 10, 2001 AFOSR

801 N. Randolph St., 11th Floor Conference Room Arlington, VA 22203-1977

0800-0830	Registration and Continental Breakfast				
0830-0840	Opening Remarks – H. Weinstock, AFOSR				
0840-0920	Exec. Summary: U. of Delaware, G. Hadjipanayis				
0920-1000	Exec. Summary: Carnegie Mellon U., M. McHenry				
1000-1020	Break				
1020-1100	AFRL Perspective and Program, R. Fingers				
1100-1130	U. of Nebraska Summary, D. Sellmyer				
1130-1200	Virginia State U. Summary, A. Arrott				
1200-1300	Lunch				
1300-1400	U. of Delaware Annual Report (various speakers)				
1400-1500	Carnegie Mellon Annual Report: D. E. Laughlin - Moderator				
	Frank Johnson				
	Amy Hsiao				
	Suk-Joon Son				
	Mei-qing Huang				
	Krishna Chowdary				
	Mitra Taheri				
1500-1520	Break				
1520-1540	U. of Nebraska (additional info), R. Skomski				
1540-1600	DARPA Metamaterials Program, V. Browning				
Discussion of DoD Magnetic Materials Programs and Future Requirements –					
	K. Hathaway, J. Prater, R. Fingers et al.				

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- Synthesis, Structure, and Properties of Carbon Encapsulated Metal Nanoparticles. M. E. McHenry and S. Subramoney, <u>Fullerenes: Chemistry</u>, <u>Physics and Technology</u>, (K. M. Kadish and R. S. Ruoff, editors) John Wiley, (2000).
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- Effects of Co Substitution on the Magnetic Properties of Pr3(Fe1-xCox)27.5Ti1.5 (x = 0-0.3), V. R. Shah, G. Markandeyulu, K. V. S.Rama Rao, M. Q. Huang, K. Sirisha, M. E. McHenry, J. Appl. Phys., Vol. 85, pp. 4678-80, (1999).
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- Structure and Thermomagnetic Properties of New FeCo-Based Nanocrystalline Ferromagnets. F. Johnson, P. Hughes, R. Gallagher, D.E. Laughlin, M.E. McHenry, M.A. Willard and V.G. Harris, IEEE Transactions on Magnetics, Vol. 37, 2261-2263, (2001).
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